Routine Use of the Varian Mercury-200 NMR Spectrometer

Nomenclature

- "VNMR" or "vnmr" refers to the nmr program that runs the spectrometer.
- "CDE" is "Common Desktop Environment", the operating system "window" that the computer boots into. It has several useful utilities.
- "Glide" is the Varian menu-driven program for taking spectra; it is typically the easiest and most restrictive way to do so.
- [Name] refers to a menu button marked "Name"; these can appear on the at the top of the screen when using Glide, just below the area reserved for typing commands (these are vnmr menu buttons) or in a pop-up menu under discussion.
- [Name1] → [Name2] means that you click on the [Name1] button to reveal the [Name2] button, which you then also click.
- [Ret] or [ret] means pressing the return key.
- VNMR commands can be strung together with spaces in between, such as
  pl, pscale, page (to plot a spectrum with x-axis scale).
- Remember, UNIX and VNMR commands are case-sensitive!

Locking and Shimming

Please note that the instrument is very sensitive to magnetic field homogeneity. Poor signal strength and false multiplets are common if shimming is not good.

Set up your experiment in Glide or by selecting parameters and typing **su** [Ret] (see "Routine Data Acquisition", below).

[Acqi] → [Lock] *turn spinning off, turn lock off, eject sample, lower new sample, turn spinning back on, turn lock back on*

If shim is likely to be way off, load standard shim parameters by typing the following vnmr commands:

rts('filename') [ret]
shim files have the same name as the solvent: "cdcl3", "c6d6", "acetone", "d2o", etc.
su [ret]

Adjust Z0, lock power, and lock gain to the values in the table below (lock power and gain can be higher if the sample is highly concentrated, has undissolved stuff floating around, or has nondeuterated solvent mixed in. Note, however, that lock power can be so high as to "saturate" the signal and reduce the lock signal. You know you are saturated if you reduce lock power and the lock level goes up.)

Solvent	Lock Power	Lock Gain	Z0
$CDCl_3$	28	29	950
C_6D_6	16	25	900
D_2O	18	26	1100?
acetone-d_6	16	25	1000?
CD_3CN			
THF-d_8			
DMSO-d_6			
pyridine-d_5			
CD_3OD			
toluene-d_8			

[Shim] A screen showing the lock level ("coarse" bar on top; "fine" bar under this) and a set of shims (Z1, Z2, Z1C, Z2C) should appear. If other shims are showing, click the toggle button marked "SHIM:" until the above four shims are shown. Adjust only Z1C and Z2C to maximize the lock level. Adjust lock gain or lock power to put the signal back on scale if it goes over 100. Note that the left mouse button accomplishes the adjustment shown on the button (-4 or –16, for example); clicking on these with the right mouse button sends the value the other way (+4 or +16). The response is pretty fast on these buttons, so you can make adjustments quickly. You can make additional shim adjustments by clicking the "SHIM:" button until Z1-Z5 shims are shown; typically, it is necessary to further adjust only Z3.

Click [Close] and you're done shimming. Note: the standard auto-shimming routine is not very good. You can do good auto-shimming (takes 3-5 minutes) by typing in the vnmr command area:

method='allzs' [Ret]
shim [Ret]
after shimming is done, type **method='z1z2'** [Ret]

To save your own shim settings for a particular type of sample that you may use again, type:

svs [Ret], and the system will prompt you for your desired shim file name.

To allow unlocked spectra to be obtained in Glide, issue the command in='no'.

Routine Data Acquisition

Click [Glide], then the big Glide [Setup] button. Select the experiment and solvent (use the right mouse button to click on the buttons to show the available choices). Be sure to type in a file name if you want to save the data in your directory on disk, and put comments in the box at the bottom. Click [Setup] to finish this window.

Click the Glide [Acquire] button. Select the parameters displayed, then click 'Do'. The machine will acquire the data. It is not possible using Glide to do something else while data is in acquisition, unless you open an entirely new vnmr session. See MGF to learn how to do this.

Alternative Method to Acquire Data Using VNMR Commands and Menu Buttons

Click [Setup]. (If this button is not visible, click [Main Menu] to find it). Then select the observe nucleus and solvent. This can be done with the two routine buttons, [H1,CDCl3] or [C13,CDCl3], or with the [Nucleus,Solvent] button for less common combinations. Certain more sophisticated experiments (DEPT, etc.) are available under the [Sequence] button.

If you want to change any parameters, such as sweep width (sw), number of scans (nt), or delay time between pulses (d1), type the parameter and value, such as: sw=4000 [ret].

Type su [ret]. This "loads" the hardware with the parameters you have selected. You will note a [Shim] button under [Setup], but the standard autoshim routines are not very good. *Note that doing an autoshim may ruin your careful manual shim!* Please do not click the [Gradient Autoshim on Z] button, since our machine does not have this excellent capability. See the notes in the above section if you want to do good autoshimming (using method='allzs').

If you have locked and shimmed manually and don't want the machine to do so, type **alock='n'** [Ret]. Then initiate the acquisition with the [Acquire] button, which offers several choices; note that if you want to do "Wft" you must first set the exponential multiplication factor lb (usually, lb = 0.1 or 0.2 for proton nmr; 1 or 2 for C13 nmr).

Routine Processing

In Glide, processing is obtained from the conveniently labeled **[Process]** button. Ignore "FID number"; input the desired line broadening value (0.1 for ^{1}H, 1 or 2 for ^{13}C, higher numbers for spectra with broader peaks). Click "process" to do weighted Fourier transform and automatic phase correction. Automatic phase correction may be separately applied by typing **aph** [Ret].

If you are not using Glide, the [Process] menu button takes you to self-explanatory buttons which you can use to see the FID, adjust weighting factors, and transform. Usually, you will use the [Weight, Transform] button. Alternatively, the "wft" command can be used.

The spectrum you see can be manipulated using the regular menu bar commands while still in Glide.

To **expand** the spectrum, click on the left limit with the left mouse button, the right limit with the right mouse button, and then [Expand] in the menubar. If the [Expand] button does not appear, click [Main Menu] → [Display] → [Interactive], and then re-set the right limit with the right mouse button.

The display can be expanded to fill the screen with the [Resize] menu button. [Flip] toggles the bottom parameter display window on and off.

To **phase**, click [Phase] on the vnmr menu. Click on the right side of the spectrum, then hold the mouse button down and adjust the phase. Then click on the left side of the spectrum, and drag to adjust phase. Go back to the right if necessary. Click the [Phase] button to end and save the changes. *Useful tip: to see the effecgt of phasing on the entire spectrum instead of just the highlighted part, type phasing=100 before you begin.*

To **adjust the vertical scale**, hold the middle mouse button down and drag the mouse up or down. Clicking above or below the baseline also adjusts the vertical scale. If you are integrating, this will adjust the integral levels.

Integration is controlled by a menubar button {[Main Menu] → [Interactive] →} that has three states: no integral, full integral (entire spectrum without breaks), and partial integral (with breaks). Integral break points are automatically set by Glide, and they are often quite good. To set your own, be in "partial integral" mode and use the following:

- remove all break points with cz command
- remove individual break points by clicking [Resets] and then putting the cursor next to the break point and pressing the right mouse button. Keep clicking that button to remove all break points.
- to insert break points: click [resets] menu button, then put cursor at desired point and click with the left button.
- To adjust the level and tilt of the integrals, click the [Lvl/Tlt] button. Then hold the left mouse button down on the spectrum and move the mouse up or down to adjust level; the right mouse similarly adjusts tilt. Click [Return] when done.
- To set an integral value, position the cursor in the integral and click [Set Int.].

Peak Picking is done by selecting: [Main Menu] → [Display] → [Interactive] → [Th] (threshold button]. Click on the spectrum with the left mouse button and move the line to the desired cutoff height. Click [Return] when done.

To **set a reference**, place the cursor on the line to be referenced. Click the [Main Menu] → [Display] → [Interactive] → [Ref] menu button and follow directions.

Printing/Plotting

Put what you want to plot on the screen. Glide provides a [Printing] button, which is easy to use. Alternatively, [Main Menu] → [Display] → [Plot] gets you a list of buttons. This is a menu of options: click on all that apply, and then click on [Page] to get everything to come out. Usually, you use [Plot], [Scale] (to plot the ppm scale), and [Peaks] (to get peak picking to print; the computer will report the number of peaks at this point. [Params] and [All Params] get printouts of the parameters on the spectrum. **Click [Page] to send these instructions to the printer** and get your printed spectrum.

These commands are also available by typing them in:

pl (plot) **pscale** (plot the scale); **pir** (put integral values below plot); **pap** (put parameters in the upper left hand corner); **ppf** (plot peak frequencies on top of spectral display); **pll** (plot line listing on the spectral display or separate page; **page** (print out the spectrum); **plcosy**(12,1.3,1) is a standard COSY-plotting command.

To plot an inset:

- display the wide-limit spectrum you want to plot
- save the current display with the "sl" command
- plot the current spectrum using line commands such as : **pl**, **pscale**, **pir**, **page**.
- place two cursors around the region you want to inset.
- type **inset** [Ret]. Move the inset into position by dragging with the right mouse. Vertical position can be changed by clicking on the cursor line with the Center button. Vertical scale of the inset is changed by clicking in the inset display with the center mouse button. The width of the spectrum is adjusted by dragging with the left mouse button.

To customize plot appearance and create .pict files for graphics export, use jdesign routine. (See "Getting Started" manual or MGF for more information). jplot is the command to plot from the plot design program.

Saving your data

If you did not specify a filename in Glide, your data has probably not been written to the hard drive. To do so, type **svf** [Ret]. The computer will prompt you for a file name (which will actually be the name of a subdirectory in your user directory containing the FID, processing parameters, etc. To load saved data back into VNMR, use the **rtf** command. Note that **svp** and **rtp** save and retrieve parameters only.

Archiving your data

Log into the NMR computer from your lab (Macintosh users will find "Fetch" particularly helpful) and copy your data files to your own media (Zip or Jaz drives are excellent). *Data will be purged from the computer at regular intervals*. Make sure you are on the NMR users' e-mail mailing list (send your e-mail address to mgfinn@scripps.edu), so that you will receive notice before your data is zapped.

Data can also be compressed for storage. All nmr data is in the form of directories containing several files; a recreation of the spectrum in VNMR requires all of these files. To transform a directory (which cannot be compressed) into a single file (which can be compressed), issue the unix commands:

> tar cvf inad.tar inad.fid (puts directory "inad.fid" into a single file "inad.tar")

> tar xvf inad.tar (restores file "inad.tar" to whatever it was created from, in this case the directory "inad.fid")

Compression can then be done with the CDE file compression utility. In File Manager, expansion of the compressed file is done by double-clicking on the file.z file.

When you are done:

- **Lock and shim the standard $CDCl_3$ sample.**
- **Always exit VNMR before logging out:**
- **Exit VNMR** by typing "exit" [Ret], or by clicking on the [Main Menu] → [More] → [Exit] button.
- **Log out** by holding the right mouse button down and selecting "Log out…"

Never Do These Two Things:

Never click the little "EXIT" button on the CDE menu bar (at the bottom of the screen) while VNMR is running.

Never shut the computer off for any reason!

If the computer hangs up, call M.G. Finn (4-8845), the Research Computing help desk (4-9369) or Stephanie Gates in Research Computing (4-9363). Other helpful numbers: Macintosh help: 4-9366 (Matt) or 4-9330 (David).

Useful Notes

VNMR commands

nt = number of scans (transients)
sw = sweep width (Hz)
dg = displays current parameters
ft = Fourier transform with no weighting
wft = weighted Fourier transform (applies "lb" value)
Note: you can set a weighting function (resolution enhancement or s/n enhancement) with the [Massage Data] button or the "wti" command.
Zero-filling is done by setting fn (number of points in Fourier transform) to 2*np (np = number of points in FID). Do this whenever you are using resolution enhancement.
cd (returns you to your home directory)

To retrieve data into VNMR: two methods exist

(1) issue the vnmr command "listenon", then open File Manager from the CDE menu bar. Find your desired data file in your directory (data files are stored in: /export/home/username/vnmrsys/data) and double-click on it.

–or–

(B) in VNMR, type **rt** [Ret]. The system will prompt you for the file name.

You can then process the data as usual.